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(56) Documents Cited

EP 0597390 A1

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#### (54) Method and apparatus for indicating optical anisotropy

(57) The optical anisotropy of a transparent birefringent sample 13 is measured by taking an assembly 10 in sequence of a primary polariser 11, the sample and a polarising analyser 14, passing monochromatic light through said assembly so it is transmitted through the sample or (Fig. 2) from a surface of the sample, rotating the plane of polarisation, using the light which has passed through the assembly to form an image of the sample, collecting the image in two dimensions for at least two rotational orientations of the plane of polarisation, and presenting the collected two-dimensional images in false colours for indications or measures across the sample of its optical anisotropy.

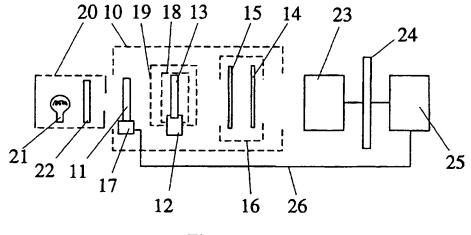


Fig. 1

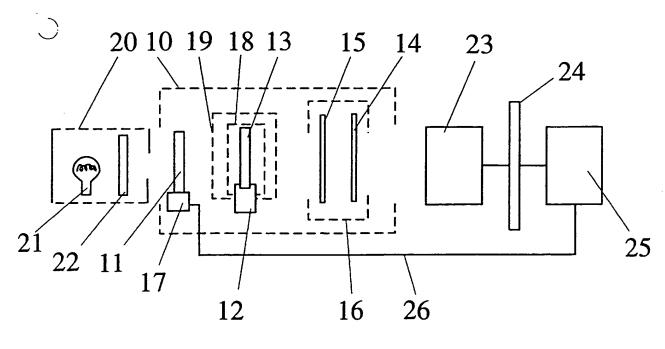
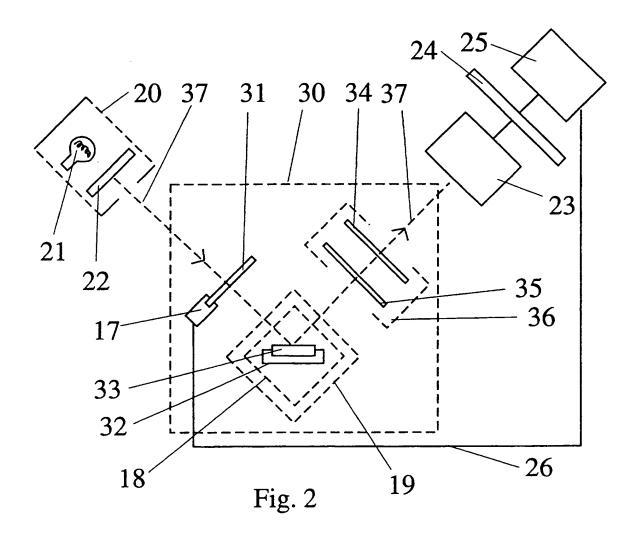


Fig. 1



## METHOD AND APPARATUS FOR INDICATING OPTICAL ANISOTROPY

This invention relates to a method and apparatus for indicating the optical anisotropy of a birefringent sample, which (as examples only) may be an anisotropic crystal, or a normally isotropic material, such as glass or methyl methacrylate sheet, which exhibits anisotropy when under strain.

A birefringent material has different refractive indices according to the planes of polarisation of light transmitted through it. The refractive indices concerned define an ellipsoid conventionally called the optical indicatrix of that material. The angular orientation of the major axis of a section of the optical indicatrix relative to a standard orientation is referred to in this specification as an indicator or measure of the optical anisotropy of a birefringent sample.

Light transmitted through a birefringent material is split into two rays which travel at different speeds through the material, thereby defining the different refractive indices referred to above and accumulating a retardation of the slower transmitted light ray relative to the faster transmitted light ray. For a particular wavelength and thickness of a sample, the relative retardation is also referred to in this specification as an indicator or measure of the optical amisotropy of a

birefringent sample and is measured by the relative length of the axes of the indicatrix of the sample.

It is an object of this invention to provide an improved method and apparatus for indicating or measuring the optical anisotropy of a birefringent sample.

the present invention, a In accordance with indicating or measuring the optical anisotropy of a transparent birefringent sample comprising taking an assembly in sequence of a primary polariser, the sample and a polarising analyser, passing monochromatic light through said assembly in the the primary polariser to the polarising direction from analyser, rotating the plane of polarisation, using the light which has passed through the assembly to form an image of the sample, collecting the image in two dimensions for at least two rotational orientations of the plane of polarisation, and presenting the collected two-dimensional images in false colours for indications or measures across the sample of its optical anisotropy.

Preferably wherein monochromatic light passing through said assembly is transmitted through the transparent birefringent sample.

Alternatively monochromatic light passing through said assembly is reflected from a surface of the transparent birefringent

sample.

Preferably also the monochromatic light is generated by a non-laser source.

Preferably also the primary polariser is a modulating crystal which is used to rotate the plane of polarisation in discrete steps or smoothly.

Alternatively, the step of rotating the plane of polarisation is achieved by physically rotating the primary polariser in discrete steps or smoothly.

Preferably also the polarising analyser is a circular polariser.

Preferably further the step of presenting the collected two-dimensional images in false colours comprises computer processing of said image and displaying the results on a computer screen.

Preferably further said computer processing enables an indication or measure of the relative retardation of the corresponding area of the sample to be displayed on each pixel or small group of pixels on the computer screen.

Preferably further said computer processing enables an

indication or measure of the relative orientation of the optical indicatrix of the anisotropy of the corresponding area of the sample to be displayed on each pixel or small group of pixels on the computer screen.

Preferably further white light is filtered to be monochromatic.

Preferably further the image is examined in two dimensions by a video camera.

Preferably further the sample is a mineral specimen or a biological sample.

The sample may be subjected to external stimulus to induce phase changes in its structure, for example by a heater/cooler or a pressure/vacuum system.

The sample may be a strained model of an object to be studied for the effects of stresses applied to the object, in which case the strained model may be a transparent layer physically applied to a face of said object.

In accordance with the present invention also, a method of indicating or measuring the optical anisotropy of a transparent birefringent sample comprises taking an assembly in sequence of a primary polariser, the sample and a polarising analyser, passing light through said assembly in either direction with

the light being reflected from a surface of the sample, rotating the plane of polarisation, using the light which has passed through the assembly to form an image of the sample, collecting the image in two dimensions for at least two rotational orientations of the plane of polarisation, and examining the collected two-dimensional images for indications or measures across the sample of its optical anisotropy.

In accordance with a further aspect of the present invention, apparatus for indicating or measuring the optical anisotropy of a transparent birefringent sample comprises an assembly in sequence of a primary polariser, a sample holder for the sample and a polarising analyser, means for passing monochromatic light through said assembly in the direction from the primary polariser to the polarising analyser, means for rotating the plane of polarisation, means for using light which has passed through the assembly to form an image of the sample, means for collecting the image in two dimensions for at least two rotational orientations of the plane of polarisation and means for presenting the collected two-dimensional images in false colours for indications or measures across the sample of its optical anisotropy.

The assembly may be arranged for monochromatic light to be transmitted through the sample when the sample is held in the sample holder, or alternatively the assembly may be arranged for monochromatic light to be reflected from a surface of the

sample when the sample is held in the sample holder.

Preferably the apparatus includes a non-laser source for the monochromatic light.

Preferably the primary polariser is a modulating crystal, and said means for rotating the plane of polarisation comprises operation means for modulating said crystal in discrete steps or smoothly.

Alternatively said means for rotating the plane of polarisation includes means for physically rotating the primary polariser in discrete steps or smoothly.

Preferably also the polarising analyser is a circular polariser.

Preferably further the circular colariser is a combination of a plane polariser and a quarter-wave plate.

Preferably further said means for presenting the collected two-dimensional images in false colours comprises a computer for processing said image and displaying the results on a computer screen.

Preferably further said means for presenting the image in two dimensions comprises a video camera.

Preferably further the sample holder comprises either a temperature-controlled environment for heating or cooling the sample, or a pressure system for pressurising the sample to induce phase changes in the structure of the sample. Alternatively, other mechanisms may be employed to apply external stimulus to the sample to induce phase changes in the structure of the sample.

In accordance with yet another aspect of the present invention, apparatus for indicating or measuring the optical anisotropy of transparent birefringent sample comprises an assembly in sequence of a primary polariser, a sample holder for the sample and a polarising analyser, means for passing light through said assembly in either direction with the light being reflected from a surface of the sample, means for rotating the plane of polarisation, means for using light which has passed through the assembly to form an image of the sample, means for collecting the image in two dimensions for at least two rotational orientations of the plane of polarisation and means for examining the collected two-dimensional images indications or measures across the sample of its optical anisotropy.

Other preferred features of the invention will be apparent from the following description and from the subsidiary claims of the specification. The invention will now be further described, merely by way of example, by reference to the accompanying drawings, in which:-

Figure 1 is a diagrammatic view of apparatus for indicating the optical anisotropy of a birefringent sample according to a first preferred example of the invention.

Figure 2 is a view similar to Figure 1 for a second preferred example of the invention.

Referring initially to Figure 1 of the accompanying drawings, there is arranged an assembly 10, in sequence, of a primary polariser 11, a sample holder 12 for a birefringent sample 13 and a polarising analyser 14. In this preferred includes a quarter-wave plate 15, sequence also the adjacent the polarising analyser 14, whereby the quarter-wave plate 15 and the polarising analyser 14 constitute a circular The primary polariser 11 is supported by polariser 16. mounting means 17 and is rotatable about an axis normal to the primary polariser 11, as more fully described below, in discrete steps by a stepper motor (not shown).

The sample holder 12 includes a temperature-controlled and temperature-monitored heater 18 for heating the sample 13 if required, and a pressure system 19 for applying pressure to the sample 13 if required. The heater 18 and/or the pressure

system 19 can be activated for crystalline or mineral samples in which phase changes are induced by temperature and/or pressure accordingly, and the changes in birefringence caused by such phase changes are very accurate indications of the temperature and/or pressure conditions that cause those phase changes to occur.

The apparatus of Figure 1 also includes a monochromatic light source 20, which is of a suitable wavelength for the quarter-wave plate 15, eg. 6238 Angstrom Units. The light source 20 is preferably not a laser, because this can suffer from "laser speckle" across the width of the sample 13. "Laser speckle" is a term given to the phenomenon in which, for a wide light beam necessary to cover the whole sample 13, the dots of a laser beam become separate across the beam width, and the eventual image becomes degraded. In this example, the monochromatic light source 20 is a white light source 21 and a monochromatic filter 22.

The assembly 10 and light source 20 are assembled on and partly constituted by a polarising microscope such as a Reichert Zetophan (trade name), as a typical example only.

The apparatus of Figure 1 further includes a charge-coupled device (CCD) video camera 23 arranged to receive the image of the sample 13 after it has passed through the circular polariser 16. The video camera 23, which may be a MVD CV960

colour camera with a half inch CCD sensor, is connected to a framegrabber 24 which is a full colour, real time image capture card. The framegrabber 24 is connected to a computer 25. The computer 25 is connected at 26 to the mounting means 17 to receive an input of the rotational orientation of the primary polariser 11.

In the method of use of this example, the sample 13 of the birefringent material to be examined is mounted on the sample holder 12, and the heater 18 or pressure system 19 activated if desired. The mounting means 17 is set into motion, rotating the primary polariser about an axis normal to the polariser in at least two steps for each measurement period to achieve corresponding rotational orientations of the plane of polarisation. In this example each step is of 7.5 degrees, and twenty-four steps total 180 degrees.

Monochromatic light from the light source 20 passes through the primary polariser 11 so that plane polarised light is incident on and passes through the birefringent sample 13. Each crystalline domain in the sample has the effects on that incident plane polarised light of defining the angular orientation of the major axis of a section of its optical indicatrix, and accumulating a retardation of the slower transmitted light ray relative to the faster transmitted light ray. The light from the sample then passes through the circular polariser 16 to provide an image of the sample 13 in

elliptically polarised light. The intensity I of the light emitted by the circular polariser 16 is given by the formula:-

 $I = I_0 / 2 [1 + \sin 2(wt - 0) \sin d]$ 

where

- Io is the incident intensity entering the sample
   (which is then affected by the absorption of the
   sample at each particular point)
- w is the angular frequency of the plane of polarisation
- t is time
- is a phase angle measured from some arbitrary position of the rotating polariser with respect to the major axis of the projected indicatrix
- d is the retardation, given by

$$d = (2\pi/\lambda) \triangle nx$$

where

is the wavelength of the monochromatic light
x is the thickness of the sample

This equation is true for each point on the sample, with the resulting intensity I of each point of the sample observed by the video camera 23 and stored by the framegrabber 24, all as a two-dimensional image of the sample 13. The formula for I given above can be used by the computer 25, with input from connection 26, to assess the intensity signal I as the primary polariser 11 rotates with time t, and at any point in the image the quantities sin d (a measure of the retardation) and some connection and some connection 23.

angular orientation of the major axis of a section of the optical indicatrix relative to a standard orientation) can be calculated separately. This is done by measuring the intensity I at each point in the image for a number of angles of the rotating polariser 11. This gives for each point in the image a set of intensities with which the computer can solve the equation for I. A Fourier decomposition method can be used for this purpose.

The result of this is that an image is plotted on the screen of the computer 25 where each pixel or small group of pixels has a value representing either sin d or fat that position. For most effective display, false colours are used to indicate those values of either sin d (related to the retardation and hence the relative birefringence) or f (related to the orientation of the indicatrix). The sin d and fimages may be recorded photographically or in a database.

Although the apparatus and its method of use in this example has the flexibility to indicate the optical anisotropy of all points on the sample by using either sin d or p above, for example as sequential images on the computer screen, it will be appreciated that the operator may choose either sin d or p alone, and operate solely with that choice.

Crystals and mineral samples have been mentioned above but the method and apparatus of this example may be used more generally

for materials science samples because transparent materials show large anisotropies due to many causes, eg. twinned crystals, defects, strains and changes in composition. Further uses are in biological microscopy where very small optical changes may be seen in transparent samples, leading to further investigation of the strains or structure causing those optical changes. For example, the optical contrast between different regions of an unstrained cell is very small, but birefringent changes across a cell can be imaged using this example of the invention in a highly effective manner.

It will be appreciated that, because the video camera 23 is collecting images of the sample 13 made up of different intensities point-by-point across each image, a monochromatic or "black and white" camera may be used instead of a colour camera.

In theory, the assembly 10 could be completely reversed, so that light from the source 20 first reaches the circular polariser 16 and then leaves the assembly through the rotating polariser 11 towards the video camera 23. All components of the apparatus would hypothetically work in the same way as described above in relation to Figure 1, the formula for the intensity I would still hold, and the computer would still function to image measures of birefringence. However any surface defects on the rotating polariser 11 would be in focus for the video camera 23 to see. Those defects would, of

course, be rotating with the rotation of the polariser 11, and this would cause noise for the computer. In contrast, in the case of the apparatus of Figure 1, the rotating polariser is not in focus and, therefore, the effects of any surface defects on the rotating polariser will be negligible.

The formula:-

$$I = I_0 / 2 \{1 + \sin 2(wt - 0) \sin d\}$$

quoted and explained above is not only applicable to the apparatus of Figure 1 and its method of use, where light is transmitted through a transparent birefringent sample, but is also applicable to apparatus and its method of use where light is reflected from the surface of a transparent birefringent sample. This application is utilised in a second preferred example of the invention which is now described with reference to Figure 2 of the accompanying drawings.

Referring to Figure 2, there is arranged an assembly 30, in sequence, of a primary polariser 31, a sample holder 32 for a birefringent sample 33 and a polarising analyser 34. The light path 37 from the primary polariser 31 to the polarising analyser 34 is arranged via reflection from a surface of a birefringent sample 33 in the sample holder 32, in contrast to the situation in the first preferred example shown in Figure 1 where the light path is via transmission through the

birefringent sample 13. However, in both preferred examples it is still true that light is passed through said assembly of primary polariser, sample and polarising analyser.

Returning specifically to the second preferred example of the invention and Figure 2, there is also a quarter-wave plate 35 adjacent the polarising analyser 34, whereby the quarter-wave plate 35 and the polarising analyser 34 constitute a circular polariser 36.

The other components are the same as those described above in the first preferred example of the invention and shown in Figure 1, have the same reference numbers as in Figure 1 and function in the same way as before.

In this example the examination of a birefringent sample by reflection provides greater flexibility in terms of the types of sample to be studied. In particular, strained Perspex models of articles or structures or other objects may be studied for the effect of stress applied to the object. The strained model may be a transparent layer, for example of methyl methacrylate sheet, physically applied to a face of an object, and then stressed as a result of the stresses applied to that object.

In a further modification of the apparatus described above in relation to Figure 1 or Figure 2 of the accompanyings drawings,

the primary polariser 11 and its mounting means 17 are changed to a modulating crystal which has operation means for modulating the crystal in discrete steps or smoothly. This provides the corresponding rotation of the plane of polarisation.

It will be appreciated that, although the mounting means 17 is described above as preferably comprising a stepper motor, a smoothly rotating motor can be used for physically rotating the primary polariser 11 or 31, or if a modulating crystal is used, this can be operated to smoothly rotate the plane of polarisation produced by the primary polariser. In this case the image of the sample 13 or 33 coming from the assembly 10 or 30 also smoothly changes, and the framegrabber 24 instantly collects images at particular orientations of the plane of polarisation under the control of the computer 25.

It will also be appreciated that if the sample is highly light-absorbing, the formula given above becomes incomplete and therefore the results will be approximate. This is true for a transparent sample through which light is transmitted, as in the first preferred example and Figure 1, and it is also true for a transparent sample from which light is reflected, as in the second preferred example and Figure 2.

#### CLAIMS

- 1. A method of indicating or measuring the optical anisotropy of a transparent birefringent sample comprising taking an assembly in sequence of a primary polariser, the sample and a polarising analyser, passing monochromatic light through said assembly in the direction from the primary polariser to the polarising analyser, rotating the plane of polarisation, using the light which has passed through the assembly to form an image of the sample, collecting the image in two dimensions for at least two rotational orientations of the plane of polarisation, and presenting the collected two-dimensional images in false colours for indications or measures across the sample of its optical anisotropy.
- 2. A method according to Claim 1 wherein monochromatic light passing through said assembly is transmitted through the transparent birefringent sample.
- 3. A method according to Claim 1 wherein monochromatic light passing through said assembly is reflected from a surface of the transparent birefringent sample.
- 4. A method according to any one of the preceding claims wherein the monochromatic light is generated by a non-laser source.

- 5. A method according to any one of the preceding claims wherein the primary polariser is a modulating crystal which is used to rotate the plane of polarisation in discrete steps or smoothly.
- 6. A method according to any one of Claims 1 5 wherein the step of rotating the plane of polarisation is achieved by physically rotating the primary polariser in discrete steps or smoothly.
- 7. A method according to any one of the preceding claims wherein the polarising analyser is a circular polariser.
- 8. A method according to any one of the preceding claims wherein the step of presenting the collected two-dimensional images in false colours comprises computer processing said image and displaying the results on a computer screen.
- 9. A method according to Claim 8 wherein said computer processing enables an indication or measure of the relative retardation of the corresponding area of the sample to be displayed in each pixel or small group of pixels on the computer screen.
- 10. A method according to Claim 8 wherein said computer processing enables an indication or measure of the relative orientation of the optical indicatrix of the anisotropy of the

corresponding area of the sample to be displayed on each pixel or small group of pixels on the computer screen.

- 11. A method according to any one of the preceding claims wherein white light is filtered to be monochromatic.
- 12. A method according to any one of the preceding claims wherein the image is examined in two dimensions by a video camera.
- 13. A method according to any one of the preceding claims wherein the sample is a mineral specimen or a biological sample.
- 14. A method according to any one of the preceding claims wherein the sample is subjected to external stimulus to induce phase changes in its structure.
- 15. A method according to Claim 3 wherein the sample is a strained model of an object to be studied for the effects of stresses applied to the object.
- 16. A method according to Claim 15 wherein the strained model is a transparent layer physically applied to a face of said object.
- 17. A method of indicating or measuring the optical anisotropy

- of a transparent birefringent sample comprising taking an assembly in sequence of a primary polariser, the sample and a polarising analyser, passing light through said assembly in either direction with the light being reflected from a surface of the sample, rotating the plane of polarisation, using the light which has passed through the assembly to form an image of the sample, collecting the image in two dimensions for at least two rotational orientations of the plane of polarisation, and examining the collected two-dimensional images for indications or measures across the sample of its optical anisotropy.
- 18. Apparatus for indicating or measuring the optical anisotropy of a transparent birefringent sample comprising an assembly in sequence of a primary polariser, a sample holder for the sample and a polarising analyser, means for passing monochromatic light through said assembly in the direction from the primary polariser to the polarising analyser, means for rotating the plane of polarisation, means for using light which has passed through the assembly to form an image of the sample, means for collecting the image in two dimensions for at least two rotational orientations of the plane of polarisation and means for presenting the collected two-dimensional images in false colours for indications or measures across the sample of its optical anisotropy.
- 19. Apparatus according to Claim 18 wherein said assembly is arranged for monochromatic light to be transmitted through the

sample when the sample is held in the sample holder.

- 20. Apparatus according to Claim 18 wherein said assembly is arranged for monochromatic light to be reflected from a surface of the sample when the sample is held in the sample holder.
- 21. Apparatus according to any one of Claims 18 20 including a non-laser source for the monochromatic light.
- 22. Apparatus according to any one of Claims 18 21 wherein the primary polariser is a modulating crystal, and said means for rotating the plane of polarisation comprises operation means for modulating said crystal in discrete steps or smoothly.
- 23. Apparatus according to any one of Claims 18 21 wherein said means for rotating the plane of polarisation includes means for physically rotating the primary polariser in discrete steps or smoothly.
- 24. Apparatus according to any one of Claims 18 23 wherein the polarising analyser is a circular polariser.
- 25. Apparatus according to Claim 24 wherein the circular polariser is a combination of a plane polariser and a quarter-wave plate.

- 26. Apparatus according to any one of Claims 18 25 wherein said means for presenting the collected two-dimensional images in false colours comprises a computer for processing said image and displaying the results on a computer screen.
- 27. Apparatus according to any one of Claims 18 26 wherein said means for presenting the image in two dimensions comprises a video camera.
- 28. Apparatus according to Claim 19 wherein the sample holder comprises a mechanism to apply external stimulus to induce phase changes in the structure of the sample.
- or measuring the optical for indicating Apparatus anisotropy of a transparent birefringent sample comprising an assembly in sequence of a primary polariser, a sample holder for the sample and a polarising analyser, means for passing light through said assembly in either direction with the light being reflected from a surface of the sample, means for rotating the plane of polarisation, means for using light which has passed through the assembly to form an image of the sample, means for collecting the image in two dimensions for at least two rotational orientations of the plane of polarisation and means for examining the collected two-dimensional images for indications or measures across the sample of its optical anisotropy.

- 30. A method of indicating or measuring the optical anisotropy of a birefringent sample substantially as hereinbefore described in relation to Figure 1 of the accompanying drawings.
- 31. A method of indicating or measuring the optical anisotropy of a birefringent sample substantially as hereinbefore described in relation to Figure 2 of the accompanying drawings.
- 32. Apparatus for indicating or measuring the optical anisotropy of a birefringent sample substantially as hereinbefore described and shown in Figure 1 of the accompanying drawings.
- 33. Apparatus for indicating or measuring the optical anisotropy of a birefringent sample substantially as hereinbefore described and shown in Figure 2 of the accompanying drawings.





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Examiner:

**David Summerhayes** 

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# Patents Act 1977 Search Report under Section 17

# Databases searched:

UK Patent Office collections, including GB, EP, WO & US patent specifications, in:

UK Cl (Ed.O): G1A (ACFX)

Int Cl (Ed.6): G01J 4/00, 4/04; G01L 1/24; G01N 21/21, 21/23

Other: ONLINE: WPI

## Documents considered to be relevant:

Category	Identity of document and relevant passage		Relevant to claims
A	EP 0597390 A1	(NAKAI et al)	
A	US 5257092	(NOGUCHI)	

Document indicating tack of novelty or inventive step Document indicating lack of inventive step if combined

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